FORM PTO-1390 (REV. 9-2001) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY 'S DOCKET NUMBER: CU-2686 WDD

U.S. APPLICATION NO. (If known, see 37 CFR 1.5): 10/030300

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/03762	26 April 2000	5 May 1999

TITLE OF INVENTION: COLLAGEN-FREE COSMETIC PREPARATIONS

APPLICANT(S) FOR DO/EO/US: Rolf WACHTER et al

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. [X] This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. [ ] This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. [X] This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. [X] The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. [ ] is attached hereto (required only if not communicated by the International Bureau).
   b. [X] has been communicated by the International Bureau.
   c. [ ] is not required, as the application was filed in the United States Receiving Office (RO/US).
6. [X] An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. [X] is attached hereto.
   b. [ ] has been previously submitted under 35 U.S.C. 154(d)(4).
7. [X] Amendments to the claims of the International Aplication under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. [ ] are attached hereto (required only if not communicated by the International Bureau).
   b. [ ] have been communicated by the International Bureau.
   c. [ ] have not been made; however, the time limit for making such amendments has NOT expired.
   d. [X] have not been made and will not be made.
8. [ ] An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. [ ] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. [ ] An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. [ ] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. [ ] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. [ ] A FIRST preliminary amendment.
14. [ ] A SECOND or SUBSEQUENT preliminary amendment.
15. [ ] A substitute specification.
16. [ ] A change of power of attorney and/or address letter.
17. [ ] A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. [ ] A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. [ ] A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. [ ] Other items or information:

Express Mail Label No. EL698182828US

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
10/030300	PCT/EP00/03762	CU-2686 WDD

21. [X] The following fees are submitted:	CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO **$1040.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO **$890.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO **$740.00**		
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) **$710.00**		
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) **$100.00**		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 890.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	$
Total claims	10 - 20 =	0	x **$18.00**	$
Independent claims	2 - 3 =	0	x **$84.00**	$
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ **$280.00**	$

TOTAL OF ABOVE CALCULATIONS =	$ 890.00	
[X] Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2. +	$ 445.00	
SUBTOTAL =	$ 445.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).	$	
TOTAL NATIONAL FEE =	$	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +	$	
TOTAL FEES ENCLOSED =	$ 445.00	
Amount to be refunded:	$	
charged:	$	

a. [X] A check in the amount of $ 445.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of $ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. [X] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 12-0400 . A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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October 19, 2001

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27193
REGISTRATION NUMBER

COLLAGEN FREE COSMETIC PREPARATIONS

Field of the Invention

The invention belongs to the field of cosmetics and concerns preparations, especially face masks, which are free of animal collagen and which are obtaind through cross-linking of chitosans in the presence of glucans.

Prior Art

Cosmetic fleeces are used as moisture masks for face and hands. Normally these preparations are manufactured based on animal collagen, wherein aqueous collagen suspensions are adjusted to a pH value in the acidic area and thereafter the water is removed by freeze-drying. Because of the continuing criticism of products from animals, there is a demand in the market for products which exclusively are manufactured with use of plant raw materials or marine raw materials. From the Japanese patent application JP-A2 Hei 6/048917 (Nagawa) beauty packs with chitosan as active component as well as organic acids and collagen as further constituents are known. Object of the Japanese patent application JP-A2 Hei 4/275207 (Nitta Gelatin) are moisture binding additives to skin cosmetic agents, which are mixtures in powder form of chitosan and collagen. Object of the German patent application DE 19643066 A1 (Henkel) is further collagen free face masks, which are obtained through cross-linking of chitosan with suitable diisocyanates or dialdehydes. These are however, with regard to their dermatological compatibility until now not fully satisfactory.

The task of the invention was therefore to make available skin cosmetic agents which on one side are free from animal collagen and on the other side are suited for manufacturing of moisture masks, especially for face and hands, and which feature immunestimulation and machinability.

Object of the invention are collagen free cosmetic preparations which can be obtained by cross-linking of swollen aqueous suspensions of chitosans and ß-(1,3) glucans with diisocyanates or dialdehydes and subsequent removal of water.

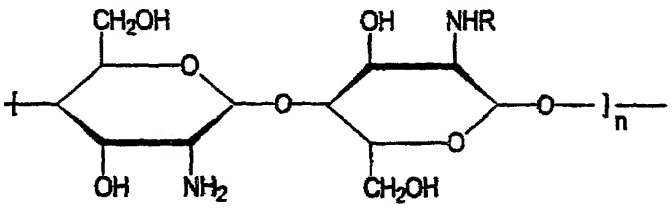
A further object of the invention concerns a method for manufacturing of collagen free cosmetic preparations, by cross-linking of swollen aqueous

suspensions of chitosans and ß-(1,3) glucans with diisocyanates or dialdehydes and subsequent removal of water.

Surprisingly it was found that addition of ß-(1,3) glucans to known cross-linked chitosans delivers cosmetic preparations, especially face masks, which have a significant better dermatological compatibility, immunestimulating effect and flexibility; at the same time the incorporation of different auxiliary substances becomes easier.

Chitosans

Chitosans are biopolymers and belong to the group of hydrocolloids. From a chemical point of view they are partial deacetylated chitins with different molecular weights, and contain the following - idealized - monomer module:



In contrast to most of the hydrocolloids, which are negatively charged in the range of biological pH-values, chitosans are under these conditions cationic biopolymers. The positively charged chitosans can interact with opposite charged surfaces and are therefore used in cosmetic hair and body care agents as well as in pharmaceutical preparations (see *Ullmann's Encyclopedia of Industrial Chemistry, 5th Ed., vol. A6,* Weinheim, Verlag Chemie, 1986, p. 231-332). A summary of these subjects are also published in for example B. Gesslein et al., *HAPPI 27,* 57 (1990), O. Skaugrud in *Drug Cosm. Ind. 148,* 24 (1991) and E. Onsoyen et al. in *Seifen-Öle-Fette-Wachse 117,* 633 (1991). By the production of chitosan chitin is used as starting material, preferably the shell residues of crust animals, which are available in large amounts as cheap raw materials. The chitin is thereby, using a method which first was described by Hackmann et al., usually first deprotonated by addition of bases, demineralized by addition of mineral acids and at last deacetylated by addition of strong bases, whereby the molecular weights can be distributed over a broad spectrum. Corresponding methods are for example known from *Makromol. Chem. 177,* 3589 (1976) or the French patent

application FR-A1 2701266. Preferably use is made of such types which are described in the German patent applications DE-A1 4442987 and DE-A1 19537001 (Henkel), and which have an average molecular weight of 10 000 to 1 200 000, preferably 40 000 to 500 000, respectively 800 000 to 1 000 000 Daltons, a viscosity according to Brookfield (1 % by weight in glycolic acid) below 5 000 mPas, a degree of deacetylation in the range of 80 to 88 % and a content of ashes of less than 0,3 % by weight.

ß-(1,3) Glucans

The term glucans means homopolysaccharides based on glucose. Depending on sterical linking there is a difference between ß-(1,3), ß-(1,4) and ß-(1,6) glucans. ß-(1,3) Glucans normally show a helical structure, whereas glucans with a (1,4) linkage generally have a linear structure. The ß-glucans of the invention have a (1,3) structure, i.e. they are substantillay free from undesired (1,6) linkages. Preferably such ß-(1,3) glucans are used where the side chains exclusively show (1,3) linkages. Especially the agents contain glucans which are obtained on the basis of yeasts from the family *Sacchaomyces*, especially *Saccharomyces cerevisiae.* Glucans of this type are available in technical amounts according to known methods. The international patent application WO 95/30022 (Biotec-Mackzymal) describes a method for producing such substances, wherein glucans with ß-(1,3) and ß-(1,6) linkages are brought in contact with ß-(1,6) glucanases in such a way, that practically all ß-(1,6) linkages are loosened. Preferably used for the manufacture of these glucans are glucanases based on *Trichodermia harzianum*. As to the manufacture and availability of the glucans contained in these agents, reference is made to the above cited publication. The glucans can be contained in the preparations in amounts of 0.1 to 5, preferably 0.2 to 5, and preferably 0.5 to 1 % by weight, based on the preparations.

Cross-linking Agents

Diisocyanates which can be used for cross-linking of the chitosans, preferably follow the formula (I),

O=CN-[X]-NC=O (I)

wherein X represents a linear or branced naphthenic or aromatic hydrocarbon residue with 1 to 12 carbon atoms. Prefreably hexametylene diisocynate is used as cross-linking agent. As dialdehydes substances following the formuls (II) can be used,

OHC-[Y]-CHO (II)

wherein Y represents a linear or branced naphthenic or aromatic hydrocarbon residue with 1 to 12 carbon atoms. Prefreably glutaric dialdehyde is used as cross-linking agent. The cross-linking agents can be used in amounts of 0.5 to 10, preferably 1 to 8, and especially 2 to 5 % by weight.

Polyols

Polyols which according to the invention can be used as further constituents in the cosmetic preparations, preferably have 2 to 15 carbon atoms and at least two hydroxyl groups. Typical examples are:

- Glycerol;
- alkylene glycols, such as for example ethylene glycol, diethylene glycol, propylene glycol, butylene glycol, hexylene glycol as well as polyethylene glycols with an average molecular weight from 100 to 1 000 Daltons;
- oligoglycerol mixtures of technical quality with a self-condensation degree of 1.5 to 10, such as e.g. technical quality diglycerol mixtures with a diglycerol content of 40 to 50 % by weight;
- methyol compounds, such as especially trimethylol ethane, trimethylol propane, trimethylol butane, pentaerythrite and dipentaerythrite;
- low alkyl glucosides, especially such with 1 to 8 carbons in the alkyl residue, such as for example methyl and butyl glucoside;
- sugar alcohols with 5 to 12 carbon atoms, such as for example sorbitol or mannit;
- sugars with 5 to 12 carbon atoms, such as for example glucose or saccharose;
- amino sugars, such as for example glucamine.

Usually the polyols are used in amounts from 1 to 10, preferably 2 to 8 % by weight, based on the dry substance oft the chitosans. Preferably use is made of glycerol and polyetylene glycols.

Manufacture of the Preparations

Normally aqueous solutions or suspensions of the chitosans with a content of dry matter of 0.5 to 3, preferably 1.8 to 2.2 % by weight with a pH value of 3.5 to 6, preferably 5.0 to 5.7 are prepared by addition of inorganic or organic acids, preferably hydrochloric acid, whereby the temperature should be chosen so that the swelling of the biopolymers is supported. Normally the temperature lies in the area from 20 to 50 and preferably 35 to 45°C. The suspensions made in this way, in addition to the dissolved biopolymers also contain swollen not dissolved particles. The viscosity of the suspension which appears through the mentioned conditions can be of influence on the mecanical properties later on. To the suspensions then the glucans and possibly polyols and further cosmetic components are added. For the mechanical properties of the fleeces it has shown to be of advantage to add to the suspension natural fibres, such as for example lignin, polyose, pektin and especially cellulose, but also sybthetic fibres such as for example polyesters, polyamides or mixtures thereof in an amount of 1 to 50, preferably 5 to 10 % by weight. It is especially recommended to add the fibres before homogenising of the solution. Subsequently the suspension is homogenised, cross-linked with the diisocyanates and/or dialdehyds, and the water is removed. Preferably the removal of water takes place through freeze-drying, and thereafter splitting into blocks or fine slices can take place.

Commercial Applicability

The preparations according to the invention are preferably used for preparation of cosmetic face masks. They can further contain as additional auxiliary and additional agents mild surfactants, oil bodies, emulsifiers, hyperfatting agents, pearl gloss waxes, consistency substances, thickening agents, polymers, silicone compounds, fats, waxes, stabilizing agents, biogenic active substances, deodorants, antitranspirants, agents against dandruff, film forming agents, swelling agents, UV light protection agents, antioxidants, hydrotropes, preservatives, insect repellents, self tanning agents, solubilizing agents, perfume oils, colouring agents and suchlike.

Typical examples of suitable mild, i.e. especially skin compatible **surfactants** are fatty alcohol polyglycol ether sulphates, monoglyceride

sulphates, mono- and/or dialkyl sulfosuccinates, fatty acid isethionates, fatty acid sarcosinates, fatty acid taurides, fatty acid glutamates, α-olefine sulphonates, ethercarboxylic acids, alkyl oligoglucosides, fatty acid glucamides, alkylamido betaines and/or protein fatty acid condensates, the last mentioned preferably based on wheat proteins.

As **oil bodies** use can be made of for example Guerbet alcohols based on fatty alcohols with 6 to 18, preferably 8 to 10 carbon atoms, esters of linear C_6-C_{22} fatty acids with linear C_6-C_{22} fatty alcohols, esters of branched C_6-C_{13} carboxylic acids with linear C_6-C_{22} fatty alcohols, such as e.g. myristyl myristate, myristyl palmitate, myristyl stearate, myristyl isostearate, myristyl oleate, myristyl behenate, myristyl erucate, cetyl myristate, cetyl palmitate, cetyl stearate, cetyl isostearate, cetyl oleate, cetyl behenate, cetyl erucate, stearyl myristate, stearyl palmitate, stearyl stearate, stearyl isostearate, stearyl oleate, stearyl behenate, stearyl erucate, isostearyl myristate, isostearyl palmitate, isostearyl stearate, isostearyl isostearate, isostearyl oleate, isosteayl behenate, isostearyl oleate, oleyl myristate, oleyl palmitate, oleyl stearate, oleyl isostearate, oleyl oleate, oleyl behenate, oleyl erucate, behenyl myristate, behenyl palmitate, behenyl stearate, behenyl isostearate, behenyl oleate, behenyl behenate, behenyl erucate, erucyl myristate, erucyl palmitate, erucyl stearate, erucyl isostearate, erucyl oleate, erucyl behenate and erucyl erucate. In additon esters of linear C_6-C_{22} fatty acids with branched alcohols, especially 2-ethylhexanol, esters of hydroxycarboxylic acids with linear or branched C_6-C_{22} fatty alcohols, especially dioctyl malate, esters of linear and/or branched fatty acids with polyvalent alcohols (such as e.g. propylene glycol, dimeric diol or trimeric triol) and/or Guerbet alcohols, triglycerides based on C_6-C_{10} fatty acids, liquid mixtures of mono-/di-/triglycerides based on C_6-C_{18} fatty acids, esters of C_6-C_{22} fatty alcohols and/or Guerbet alcohols with aromatic carboxylic acids, especially benzoic acid, esters of C_2-C_{12} dicarboxylic acids with linear or branched alcohols with 1 to 22 carbon atoms or polyols with 2 to 10 carbon atoms and 2 to 6 hydroxyl groups, plant oils, branched primary alcohols, substituted cyclohexanes, linear and branched C_6-C_{22} fatty alcohol carbonates, Guerbet carbonates, esters of benzoic acid with linear and/or branched C_6-C_{22} alcohols (e.g. Finsolv® TN), linear or branched, symmetrical or unsymmetrical dialkyl ethers with 6 to 22 carbon atoms in each alkyl group, ring opening products of epoxydated fatty acid esters with polyols, silicone oils and/or

aliphatic or naphthenic hydrocarbons, such as e.g. squalan, squalen or dialkyl cyclohexanes, can be used

As **emulsifiers** for example nonionic surfactants from at least one of the following groups may be used:

(1) Addition products of 2 to 30 moles ethylene oxide and/or 0 to 5 moles propylene oxide on linear fatty alcohols with 8 to 22 C atoms, on fatty acids with 12 to 22 C atoms and on alkyl phenols with 8 to 15 C atoms in the alkyl group;

(2) $C_{12/18}$ fatty acid mono- and diesters of addition products of 1 to 30 moles ethylene oxide and glycerol;

(3) glycerol mono- and diesters and sorbitan mono- and diesters of saturated and unsaturated fatty acids with 6 to 22 carbon atoms and their ethylene oxide addition products;

(4) alkyl mono- and oligoglycosides with 8 to 22 carbon atoms in the alkyl group and their ethoxylated analogues;

(5) addition products of 15 to 60 moles ethylene oxide on ricinus oil and/or hardened ricinus oil;

(6) polyol and especially polyglycerol esters,

(7) addition products of 2 to 15 moles ethylene oxide on ricinus oil and/or hardened ricinus oil;

(8) partial esters based on linear, branched, unsaturated or saturated $C_{6/22}$ fatty acids, ricinolic acid and 12-hydroxy stearic acid and glycerol, polyglycerol, pentaerythrite, dipentaerythrite, sugar alcohols (e.g. sorbitol), alkyl glucosides (e.g. methyl glucoside, butyl glucoside, lauryl glucoside) as well as polyglucosides (e.g. cellulose);

(9) mono-, di- and trialkylphosphates as well as mono-, di- and/or tri-PEG alkyl phosphates and their salts;

(10) wool wax alcohols;

(11) polysiloxane/polyalkyl/polyether copolymers or corresponding derivatives;

(12) mixed esters of pentaerythrite, fatty acids, citric acid and fatty alcohol according to DE 1165574 PS and/or mixed esters of fatty acids with 6 to 22 carbon atoms, methyl glucose and polyols, preferably glycerol or polyglycerol,

(13) polyalkylene glycols, as well as

(14) glycerol carbonate.

The addition products of ethylene oxide and/or of propylene oxide on fatty alcohols, fatty acids, alkyl phenols, glycerol mono- and diesters as well as sorbitan mono- and -diesters of fatty acids or on ricinus oil are known products which are commercially available. They are mixtures of homologous substances, with an average degree of alkoxylation corresponding to the ratio of the amounts of the substances ethylene oxide and/or propylen oxide and substrate, with which the addition reaction is carried out. $C_{12/18}$ fatty acid mono- and diesters of addition products of ethylene oxide on glycerol are known from DE 2024051 PS as revertive fatting agents for cosmetic preparations.

$C_{8/18}$ alkyl mono- and oligoglycosides, their manufacture and their use is known from prior art. Their preparation can especially be carried out by reaction of glucose or oligosaccharides with primary alcohols having 8 to 18 C atoms. With regard to the glycoside residue both monoglycosides, where a cyclic sugar group is glycosidic bond to the fatty alcohol, and oligomeric glycosides with a degree of oligomerisation until preferably about 8, are suitable. The degree of oligomerization is then a statistical mean value, based on a distribution of homologous which is usual for such products of technical quality.

Typical examples of suitable polyglycerol esters are polyglyceryl-2-dipolyhydroxy stearate (Dehymulus® PGPH), polyglycerol-3-diisostearate (Lameform® TGI), polyglyceryl-4-isostearate (Isolan® GI 34), polyglyceryl-3-oleate, diisostearoyl polyglyceryl-3-diisostearate (Isolan® PDI), polyglyceryl-3 metyyl cellulose diisostearate (Tego Care® 450), polyglyceryl-3 beeswax (Cera Bellina®), polyglyceryl-4 caprate (Polyglycerol caprate T2010/90), polyglyceryl-3 cetyl ether (Chimexane® NL), polyglyceryl-3 distearate (Cremophor® GS 32) and polyglyceryl polyricine oleate (Admul® WOL 1403), polyglyceryl dimerate isostearate, as well as their mixtures.

Zwitterionic surfactants can also be used as emulsifiers. The term zwitterionic surfactants is intended to mean such surface active compounds which in their molecule have at least a quaternary ammonium group and at least one carboxylate and one sulphonate group. Especially suitable zwitterionic surfactants are the so-called betaines such as the N-alkyl-N,N-dimethyl ammonium glycinates, for example the coco alkyldimethyl ammonium glycinate, N-acylaminopropyl-N,N-dimethyl ammonium glycinate, for example the coco

acylaminopropyl dimethyl ammonium glycinate, and 2-alkyl-3-carboxylmethyl hydroxyethyl imidazoline with in each case 8 to 18 C atoms in the alkyl or acyl - groups, as well as the coco acylaminoethyl hydroxyethyl carboxymethyl glycinate. Especially preferred is that under the CTFA term *cocamidopropyl betaine* known fatty acid amide derivative. Also suitable emulsifiers are ampholytic surfactants. Ampholytic surfactants are such surface active compounds which in addition to a $C_{8/18}$ alkyl or acyl group in the molecule at least contain a free amino group and at least one -COOH or -SO_3H group and which can form inner salts. Examples of suitable ampholytic surfactants are N-alkyl glycines, N-alkyl propionic acids, N-alkyl aminobutyric acids, N-alkyl iminodipropionic acids, N-hydroxyethyl-N-alkylamidopropyl glycines, N-alkyltaurines, N-alkylsarcosines, 2-alkylaminopropionic acids and alkylamino acetic acids with in each case about 8 to 18 C atoms in the alkyl group. Especially preferable ampholytic surfactants are the N-coco alkylamino propionate, the coco acylamino ethylamino propionate and the $C_{12/18}$ acylsarcosine. In addition to the ampholytic, also quaternary emulsifiers can be used, of which ester salts of the type of esterquats, preferably methylquaternised di-fatty acid triethanolamine ester salts, are especially preferable.

As **hyperfatting** agents substances such as for example lanolin and lecithin as well as polyethoxylated or acylated lanolin and lecithin derivatives, polyol fatty acid esters, monoglycerides and fatty acid alkanolamides can be used, whereby the last mentioned at the same time act as foam stabilisers.

As exemplary **pearl gloss waxes** the following should be mentioned: Alkylene glycolester, especially ethylene glycol distearate; fatty acid alkanolamides, especially coco fatty acid diethanolamide; partial glycerides, especially stearic acid monoglyceride; esters of polyvalent, possibly hydroxysubstituted carboxylic acids with fatty alcohols with 6 to 22 carbon atoms, especially long chain esters of tartaric acid; fat substances, such as for example fatty alcohols, fatty ketones, fatty aldehydes, fatty ethers and fatty carbonates, wherein the sum of carbon atoms is at least 24, especially lauron and distearyl ethers; fatty acids such as stearic acid, hydroxystearic acid or behenic acid, ring opening products of olefine epoxides with 12 to 22 carbon atoms with fatty alcohols with 12 to 22 carbon atoms and/or polyols with 2 to 15 carbon atoms and 2 to 10 hydroxyl groups as well as their mixtures.

As **consistency givers** preferably use is made of fatty alcohols or hydroxy fatty alcohols with 12 to 22 and preferably 16 to 18 carbon atoms and additionally partial glycerides, fatty acids or hydroxy fatty acids. A combination of these substances with alkyl oligoglucosides and/or fatty acid-N-methyl glucamides with the same chain length and/or polyglycerol-poly-12-hydroxy stearates is preferred.

Suitable **thickening agents** are for example types of aerosil (hydrophilic silicic acids), polysaccharides, especially xanthan gum, guar-guar, agar-agar, alginates and methyl celluloses, carboxymethyl celluloses and hydroxyethyl cellulose, as well as higher molecular polyethylene glycol mono- and diesters of fatty acids, polyacrylates, (e.g. Carbopols® from Goodrich or Synthalenes® from Sigma), polyacrylamides, polyvinyl alcohol and polyvinyl pyrrolidone, surfactants such as for example ethoxylated fatty acid glycerides, ester of fatty acids with polyols such as for example pentaerythrite or trimethylolpropane, fatty alcohol ethoxytates with narrow distribution of homologous, or alkyl oligoglucosides as well as elektrolytes such as sodium chloride and ammonium chloride.

Suitable **cationic polymers** are for example cationic cellulose derivatives, such as e.g. a quaternized hydroxyethyl cellulose, which is available under the name of Polymer JR 400® from Amerchol, cationic starch, copolymers of diallyl ammonium salts and acrylamides, quaternized vinyl pyrrolidone/vinyl imidazol polymers, such as e.g. Luviquat® (BASF), condensation products of polyglycols and amines, quaternized collagen polypeptides, such as for example lauryl dimonium hydroxypropyl hydrolyzed collagen (Lamequat®L / Grünau), quaternized wheat polypeptides, polyethylene imine, cationic silicone polymers, such as e.g. amidomethicones, copolymers of adipic acid and dimethylamino hydroxypropyl diethylenetriamine (Cartaretine® / Sandoz), copolymers of acrylic acid with dimethyl diallylammonium chloride (Merquat® 550 /Chemviron), polyamino polyamides, such as e.g. described in FR 2252840 A, as well as their cross-linked water soluble polymers, cationic chitin derivatives such as for example quaternized chitosan, possibly microcrystalline distributed, condensation products of dihalogen alkyls, such as e.g. dibromobutane with bisdialkylamines, such as e.g. bis-dimethylamino-1,3-propane, cationic guar-gum, such as e.g. Jaguar® CBS, Jaguar® C-17, Jaguar® C-16 from Celanese, quaternised ammonium salt polymers, such as e.g. Mirapol® A-15, Mirapol® AD-1, Mirapol® AZ-1 from Miranol.

As exemplary **anionic, zwitterionic, amphoteric and non-ionic polymers** the following can be used: Vinyl acetate/crotonic acid copolymers, vinyl pyrrolidone/vinyl acrylate copolymers, vinyl acetate/butyl maleate/isobornyl acrylate copolymers, methyl vinylether/maleic acid anhydride copolymers and their esters, non-cross-linked and with polyols cross-linked polyacrylic acids, acrylamido propyltrimethyl ammonium chloride/acrylate copolymers, octylacrylamide/methyl methacrylate/ tert.-butylaminoethyl methacrylate/2-hydroxypropyl methacrylate copolymers, polyvinyl pyrrolidone, vinyl pyrrolidone/ vinylacetate copolymers, vinyl pyrrolidone/ dimethylamino ethylmethacrylate/vinyl caprolactam terpolymers as well as possibly derivatized cellulose ethers and silicones.

Suitable **silicon compounds** are for example dimethyl polysiloxane, methylphenyl polysiloxane, cyclic silicones as well as amino, fatty acid, alcohol, polyether, epoxy, fluorine, glykoside and/or alkyl modified silicone compounds, which at room temperature can be in the liquid as well as in the resin state. Further suitable are simethicones, which are mixtures of dimethicones with an average chain length of 200 to 300 dimethyl siloxane units and hydrogenated silicates. A detailed survey of suitable volatile silicones can also be found in Todd et al., *Cosm.Toil. 91, 27 (1976).*

Typical exemplary **fats** are glycerides, and as **waxes** natural waxes, among others, can be used, such as e.g. candelilla wax, carnauba wax, Japan wax, espartogras wax, cork wax, guaruma wax, rice seed oil wax, sugar cane wax, ouricury wax, montan wax, beeswax, schellac wax, spermaceti, lanolin (wool wax), bürzel fat, ceresin, ozokerit (terrestrial wax), petrolatum, paraffin waxes, micro waxes; chemically modified waxes (hard waxes), such as e.g. montanester waxes, sasot waxes, hydrogenated yoyoba waxes as well as synthetic waxes, such as e.g. polyalkylene waxes and polyethylene glycol waxes.

As **stabilizers** metal salts of fatty acids, such as e.g. magnesium, aluminium and/or zinc stearate or ricinoleate can be used.

As **biogenic active substances** should be understood for example tocopherol, tocopherol acetate, tocopherol palmitate, ascorbic acid, desoxy ribonucleic acid, retinol, bisabolol, allantoin, phytantriol, panthenol, AHA acids, amino acids, ceramides, pseudoceramides, essential oils, extracts of plants and

marine organisms, vitamin complexes and biotechnological substances, such s e.g. ß-glucans or other yeast components.

Cosmetic **deodorants** act against body odours, mask them or eliminate them. Body odours develop through the effect of skin bacterias on apocrinic sweat, whereby unpleasant smelling degradation products are formed. According to this deodorants contain active substances which acts as germ inhibitors, enzyme inhibitors, odour inhibitors or odour masking agents.

As **germ inhibiting agents** principally all substances with specific effects against gram-positive bacteria, such as e.g 4-hydroxy benzoic acid and its salts and esters, N-(4-chlorophenyl)-N'-(3,4-dichlorophenyl) urea. 2,4,4'-trichloro-2'-hydroxy diphenylether, (Triclosan), 4-chloro-3,5-dimethyl phenol, 2,2'- methylen bis(6-bromo-4-chlorophenol), 3-methyl-4-(1-methylethyl) phenol, 2-benzyl-4-chlorophenol, 3-(4-chlorophenoxy)-1,2-propanediol, 3-iodo-2-propinyl butyl carbamate, chlorohexidin, 3,4,4'-trichlorocarbanilide (TCC), antibacterial odour substances, thymol, menthol, mint oil, farnesol, phenoxy ethanol, glycerol monolaurate (GML), diglycerol monocaprinate (DMC), salicylic acid n-octylamide or salicylic acid n-decylamide.

As **enzyme inhibitors** are for example esterase inhibitors suited. These are preferably trialkyl citrates such as wie trimethyl citrate, tripropyl citrate, triisopropyl citrate, tributyl citrate and especially triethyl citrate (Hydageen® CAT, Henkel KGaA, Düsseldorf/FRG). The substanses inhibit the enzyme activity and thereby reduce the odour formation. Furter substances which can be used as esterase inhibitors are sterol sulphates or phosphates, such as for example lanosterol, cholesterol, campesterol, stigmasterol and sitosterol sulphate or phosphate, dicarbonic acids and their esters, such as for example glutaric acid, glutaric acid monoethyl ester, glutaric acid diethyl ester, adipic acid, adipic acid monoethyl ester, adipic acid diethyl ester, malonic acid and malonic acid diethyl ester, hydroxycarboxylic acids and their esters such as for example citric acid, malic acid, tartaric acid or tartaric acid diethyl ester, and zinc glycinate.

As **odour absorbers** such substances are suited which take up oudour forming compounds and are able to hold them extensively. They reduce the partial pressure of each component and thereby also reduce their spreading rate. It is in this connection important that the perfumes are not affected. Odour absorber have no effect on bacterias. They contain e.g. as main component a

complexe zinc salt of ricinolic acid or special, to a high degree odour neutral scent substances which are known by the skilled person an "Fixateure", such as e.g. extracts of labdanum or styrax or certain abietinic acid derivatives. As odour masking substances odouriferous substances or parfüme oils are used, which in addition to their function as odour masking subatances give the deodorants their special scent. As perfume oils mixtures of natural and synthetic scent substances should be mentioned. Natural scent substances are extracts of flowers, stems and blades, fruits, fruit shells, roots, wood, herbs and grass, needles and twigs, as well as resins and balsams. Raw materials from animals are also possible, such as for example zibet and castoreum. Typical synthetic odour compounds are products from types of esters, ethers, aldehydes, ketones, alcohols and hydrocarbons. Odour compounds from types of esters are e.g. benzyl acetate, p-tert.-butylcyclohexyl acetate, linalyl acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, allylcyclohexyl propionate, styrallyl propionate and benzyl salicylate. Benzylethyl ether belongs for example to the ethers, to the aldehydes e.g. the linear alkanals with 8 to 18 carbon atoms, citral, citronellal, citronellyl oxyacetaldehyde, cyclamen aldehyde, hydroxy citronellal, lilial and bourgeonal, to the ketones e.g. the ionones and methylcedryl ketone, to the alcohols anethol, citronellol, eugenol, isoeugenol, geraniol, linalool, phenylethyl alcohol and terpineol; to the hydrocarbons mainly the terpenes and balsams belong. However, mixtures of different odour substances are preferred, which together give a pleasant smell. Also etheral oils with low volatility, which often are used as aroma components, are suited as perfume oils, e.g. sage oil, chamomile oil, carnation oil, melissa oil, mint oil, cinnamon leaf oil, limeflower oil, juniper berry oil, vetiver oil, oliban oil, galbanum oil, labdanum oil and lavandin oil. Preferably used are bergamot oil, dihydromyrcenol, lilial, lyral, citronellol, phenylethyl alcohol, α-hexylcinnamon aldehyde, geraniol, benzyl aceton, cyclamen aldehyde, linalool, boisambrene forte, ambroxane, indol, hedione, sandelice, lemon oil, mandarin oil, orange oil, allylamyl glycolate, cyclovertal, lavandine oil, muskateller sage oil, ß-damascone, geranium oil bourbon, cyclohexyl salicylate, vertofix coeur, iso-E-super, Fixolide NP, Evernyl, iraidein gamma, phenylacetic acid, geranyl acetate, benzyl acetate, rose oxide, romillate, irotyl and floramate, alone or in mixtures.

Antitranspirants (antiperspirants) reduce the formation of sweat through influence on the activity of the eccrine sweat glands, and therefore counteract

axillary wetness and body odour. Aqueous or water free formulations of antitranspirants typically contain the following ingredients:

(a) astringent active substances,

(b) oil components,

(c) nonionic emulsifiers,

(d) co-emulsifiers,

(e) consistency substances,

(f) auxiliaries such as e.g. thickening agents or complexing agents and/or

(g) non-aqueous solvents such as e.g. ethanol, propylen glycol and/or glycerol.

As astringent antitranspirant active substances above all salts of aluminium, zirkonium or zinc are suited. Such suitable antihydrotic active agents are e.g. aluminium chloride, aluminium chlorohydrate, aluminium dichlorohydrate, aluminium sesquichlorohydrate and their complexes, e.g. with propylene glycol-1,2. Aluminium hydroxy allantoinat, aluminium chloride tartrate, aluminium-zirkonium trichlorohydrate, aluminium-zirkonium tetrachloro hydrate, Aluminium-zirkonium pentachloro hydrate and their complexes, e.g. with amino acids such as glycine. In addition antitranspirants can contain small amounts of common oil solube and water soluble auxiliaries. Such oil solube auxiliaries can e.g. be:

- Inflammation inhibiting, skin protecting, or fragrant etheral oils,
- synthetic skin protecting active agents and/or
- oil soluble perfums.

Common water soluble additives are e.g. preservatives, water soluble scents, agents for adjustment of pH, e.g. buffer mixtures, water soluble thickeners, e.g. water soluble natural and synthetic polymers such as e.g. xanthan gum, hydroxyethyl cellulose, polyvinyl pyrrolidone or high molecular polyethylene oxides.

As **anti dandruff** agents climbazol, octopirox and zinc pyrethion can be used.

Useable **film formation** agents are for example chitosan, microcrystalline chitosan, quaternary chitosan, polyvinyl pyrrolidone, vinyl pyrrolidone/vinylacetate copolymers, polymers of the acrylic acids, quaternary derivatives of cellulose, collagen, hyaluronic acid or its salts and similar compounds.

As **swelling agents** for aqueous phases montmorillonite, clay mineral substances, pemulen, as well as alkylmodified Carbopol types (Goodrich) can be

used. Further suitable polymers or swelling agents can be found in the survey of R. Lochhead in *Cosm.Toil.108, 95 (1993)*.

UV light protection factors are e.g organic substances (light protection filters) which at room temperature are in liquid or crystalline form, and which are capable of absorbing ultraviolet radiation and to set free the received energy in the form of radiation with long wavelength, e.g. in the form of heat. UVB filters can be soluble in oils or in water. As oil soluble substances the following are mentioned as examples:

- 3-Benzyliden camphor, respectively 3-benzylidene norcamphor and the derivatives thereof, e.g. 3-(4-methylbenzylidene) camphor as described in EP-B1 0693471;
- 4-aminobenzoic acid derivatives, preferably 4-(dimethylamino) benzoic acid 2-ethylhexyl ester, 4-(dimethylamino) benzoic acid 2-octyl ester and 4-(dimethylamino) benzoic acid amyl ester;
- esters of cinnamonic acid, preferably 4-methoxy cinnamonic acid 2-ethylhexyl ester, 4-methoxy cinnamonic acid propyl ester, 4-methoxy cinnamonic acid isoamyl ester, 2-cyano-3,3-phenyl cinnamonic acid 2-ethythexylester (octocrylene);
- esters of salicylic acid, preferably salicylic acid 2-ethylhexylester, salicylic acid 4-isopropyl benzylester, salicylic acid homomenthylester;
- derivatives of benzophenone, preferably 2-hydroxy-4-methoxy benzophenone, 2-hydroxy-4-methoxy-4'-methyl benzophenone, 2,2'-dihydroxy-4-methoxy benzophenone;
- esters of benzalmalonic acid, preferably 4-methoxy benzmalonic acid 2-ethylhexyl ester,
- triazine derivatives, such as e.g. 2,4,6-trianilino-(p-carbo-2'-ethyl-1'-hexyloxy)-1,3,5-triazine and octyl triazone, as described in EP A1 0818450, or dioctyl butamido triazone (Uvasorb® HEB);
- propane-1,3-diones, such as e.g.1-(4-tert.-butylphenyl)-3-(4'-methoxy-phenyl)-propane-1,3-dion;
- ketotricyclo(5,2,1,0)-decane derivatives, as described in EP 069521 B1.

As water soluble substances the following can be mentioned:

- 2-Phenylbenzimidazol-5-sulphonic acid and the alkali, alkaline earth, ammonium, alkylammonium, alkanolammonium and glucammonium salts;

- sulphonic acid derivatives of benzophenones, preferably 2-hydroxy-4-methoxybenzophenon-5-sulphonic acid and their salts;
- sulphonic acid derivatives of 3-benzyliden camphor, such as e.g. 4-(2-oxo-3-bornylidenemethyl)-benzene sulphonic acid and 2-methyl-5-(2-oxo-bornylidene) sulphonic acid and their salts.

As typical UV-A filters especially derivatives of benzoyl methane come in question, such as e.g. 1-(4'-tert.-butylphenyl)-3-(4'-methoxyphenyl)propane-1,3-dion, 4-tert.butyl-4'-methoxydibenzoyl-methane (Parsol 1789), or 1-phenyl-3-(4'-isopropylphenyl-propane-1,3-dion, as also enamine compounds, as described in DE 19712033 (BASF). The UV-A and UV-B filters can of course also be used in mixtures. In addition to the mentioned soluble substances also insoluble light protection pigments can be used for this purpose, i.e. fine disperse metal oxides or salts. Examples of suitable metal oxides are especially zinc oxide and titanium dioxide and in addition other oxides of iron, zirconium, silicon, manganese, aluminium and cerium, as well as their mixtures. As salts silicates (talk), barium sulphate or zinc stearate can be used. The oxides and salts are used in the form of the pigments for skin caring and skin protecting emulsions and decorative cosmetics. The particles should have an average diameter of less than 100 nm, preferably between 5 and 50 nm and especially between 15 and 30 nm. They can have a spherical shape, but particles can also be used which have an ellipsoidal form or else have a shape which differs from the spherical shape. The pigment can also be present in a surface treated form, i.e. made hydrophilic or hydrophobic. Typical examples are coated titanium dioxides, such as e.g. Titandioxid T 805 (Degussa) or Eusolex® T2000 (Merck). As hydrophobic coating agents preferably silicones and especially trialkoxy octyl silane or Simethicone can be used. In sun protecting agents preferably so-called micro or nano pigments are used. In sun protecting agents preferably so-called micro or nano pigments are used. Preferably micronized zinc oxide is used.

Further suitable UV light protection factors can be found in the survey by P.Finkel in *SÖFW-Journal 122, 543 (1996).* In addition to the primary light protection substances also secondary light protection substances of the **antioxidant** type find use, which interrupt the photochemichal reaction chain, which is initiated when UV radiation penetrates the skin. Typical examples of such are amino acids (e.g. glycine, histidin, tyrosin, tryptophan) and their derivatives,

imidazoles (e.g. urocaninic acid) and their derivatives, peptides such as D,L-camosine, D-camosine, L-camosine and their derivatives (e.g. anserine), carotinoides, carotine (e.g. α-carotin, ß-carotin, lycopin) and their derivatives, chlorogenic acid and its derivatives, liponic acid and its derivatives (e.g. dihydroliponic acid), aurothioglucose, propylthiouracil and other thiols (e.g. thioredoxin, glutathion, cystein, cystin, cystamine and their glycosyl, n-acetyl, methyl, ethyl, propyl, amyl, butyl and lauryl, palmitoyl, oleyl, γ-linoleyl, cholesteryl and glyceryl esters) as well as their salts, dilauryl thiodipropionate, distearyl thiodipropionate, thiodipropionic acid and their derivatives (esters, ethers, peptides, lipides, nucleotides, nucleosides and salts) as well as sulfoximine compounds (e.g. buthionin sulfoximines, homocystein sulfoximines, butionin sulfones, penta-, hexa-, hepta-thionin sufoximine) in very small compatible doses (e.g. pmol to µmol/kg), further (metal) chelating agents (e.g. α-hydroxy fatty acids, palmitic acid, phytinic acid, lactoferrine), α-hydroxy acids (e.g. citric acid, lactic acid, malic acid), humin acid, bile acid, bile extracts, bilirubin, bifiverdin, EDTA, EGTA and their derivatives, unsaturated fatty acids and their derivatives (e.g. γ-linolenic acid, linolic acid, oleic acid), folic acid and their derivatives, ubichinon and ubichinol and their derivatives, vitamin C and derivatives (e.g. ascorbyl palmitate, Mg-ascorbyl phosphate, ascorbyl acetate), tocopherols and derivatives (e.g. vitamin E acetate), vitamin A and derivatives (vitamin A patmitate) as well as koniferyl benzoate of benzoe resin, rutinic acid and their derivatives, α-glycosylrutin, ferula acid, furfuryliden glucitol, carnosine, butylhydroxy toluene, butylhydroxy anisol, nordihydro guajak resin acid, nordihydro guajaret acid, trihydroxy butyrophenon, uric acid and their derivatives, mannose and its derivatives, super oxide dismutase, zinc and its derivatives (e.g. ZnO, $ZnSO_4$), selen and its derivatives (e.g. selen-methionin), stilbenes and their derivatives (e.g. stilbene oxide, trans-stilbene oxide), and the derivatives suitable according to the invention (salts, esters, ethers, sugars, nucleotides, nucleosides, peptides and lipids) of these mentioned active substances.

As **preservatives** for example phenoxyethanol, formaldehyde solution, parabene, pentanediol or sorbic acid as well as those mentioned in enclosure 6, parts A and B of the cosmetic regulations, are further classes of substances. As **insect repellents** N,N-diethyl-m-toluamide, 1,2-pentanediol or insect repellent 3535 come into question, as **self tanning agent** dihydroxy acetone is suited.

As **perfume oils** mixtures of natural and synthetic scent substances should be mentioned. Natural odour substances are extracts of flowers (lilies, lavendel, roses, jasmin, neroli, ylang-ylang), stems and blades (geranium, patchouli, petitgrain), fruits (anis, coriander, caraway, juniper), fruit shells (bergamot, lemon, orange), roots (macis, angelica, celery, kardamon, costus, iris, calmus), wood (stone pine, sandel, guajac, cedar, rosewood), herbs and grass (tarragon, lemongrass, sage, thyme), needles and twigs (spruce, fir, pine, traipsed), resins and balsams (galbanum, elemi, benzoe, myrrh, olibanum, opoponax). Raw materials from animals are also possible, such as for example zibet and castoreum. Typical synthetic odour compounds are products from types of esters, ethers, aldehydes, ketones, alcohols and hydrocarbons. Odour compounds from types of esters are e.g. benzyl acetate, phenoxyethyl isobutyrate, p-tert.-butylcyclohexyl acetate, linalyl acetate, dimethylbenzyl carbinyl acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, ethylmethylphenyl glycinate, allylcyclohexyl propionate, styrallyl propionate and benzyl salicylate. Benzylethyl ether belongs for example to the ethers, to the aldehydes e.g. the linear alkanales with 8 to 18 carbon atoms, citral, citronellal, citronellyl oxyacetaldehyde, cyclamen aldehyde, hydroxy citronellal, lilial and bourgeonal, to the ketones e.g. the ionones, α-isomethyl ionon and methylcedryl ketone, to the alcohols anethol, citronellol, eugenol, isoeugenol, geraniol, linalool, phenylethyl alcohol and terpineol; to the hydrocarbons mainly the terpenes and balsams belong. However, mixtures of different odour substances are preferred, which together give a pleasant smell. Also etheral oils with low volatility, which often are used as aroma components, are suited as perfume oils, e.g. sage oil, chamomile oil, carnation oil, melissa oil, mint oil, cinnamon leaf oil, limeflower oil, juniper berry oil, vetiver oil, oliban oil, galbanum oil, labolanum oil and lavandin oil. Preferably used are bergamot oil, dihydromyrcenol, lilial, lyral, citronellol, phenylethyl alcohol, α-hexylcinnamon aldehyde, geraniol, benzyl acetone, cyclamen aldehyde, linalool, boisambrene forte, ambroxane, indol, hedione, sandelice, lemon oil, mandarin oil, orange oil, allylamyl glycolate, cyclovertal, lavandine oil, muskateller sage oil, ß-damascone, geranium oil bourbon, cyclohexyl salicylate, Vertofix coeur, iso-E-super, Fixolide NP, Evemyl, iraidein gamma, phenylacetic acid, geranyl acetate, benzyl acetate, rose oxide, romillate, irotyl and floramate, alone or in mixtures.

As **colouring agents** such substances which are suited and approved for cosmetic purposes can be used, such as for example those mentioned in the publication *"Kosmetische Färbemittel" (cosmetic dyes) of the "Farbstoffkommission der Deutschen Forschungsgemeinschaft"*, published by Verlag Chemie, Weinheim, 1984, p. 81-106. These dyes are generally used in concentrations from 0.001 to 0.1 % by weight, based on the whole mixture.

The full amount of auxiliary and additional agents can be 1 to 50, preferably 5 to 40 % by weight, based on the agents.

Examples

Example 1

Into a 2 liter apparatus with a stirrer 1960 ml of water was added and wamed up to 40°C, and 40 g chitosan (Hydagen® CMPF, Henkel KGaA, Düsseldorf / FRG) was added. The pH value of the mixture was adjusted to 5.5 by addition of hydrochloric acid. Thereafter 2 g (5 % by weight based on dry substance) glycine and 0.5 g betaglucan (Higcareen® GS) was added and the mixture was homogenised with an Ultraturrax. Thereafter 0.8 g (2 % by weight based on dry substance) hexamethylene diisocyanate was carefully stirred in. After the cross-linking the suspension was frozen into a block and subsequently lyophilized. By splitting of the blocks after water removal to the desired thickness, water soluble fleeces were obtained, which by moistening behaved like sponges.

Example 2

Into a 2 liter apparatus with a stirrer 1960 ml of water was added and wamed up to 40°C, and 40 g chitosan (Hydagen® CMPF, Henkel KGaA, Düsseldorf / FRG) was added. The pH value of the mixture was adjusted to 5.5 by addition of hydrochloric acid. Thereafter 2 g (5 % by weight based on dry substance) glycine, 1 g betaglucan (Higcareen® GS) and 2 g (5 % by weight based on dry substance) cellulose fibers were added and the mixture was homogenised with an Ultraturrax. Thereafter 0.8 g (2 % by weight based on dry substance) hexamethylene diisocyanate was carefully stirred in. After the cross-linking the suspension was frozen into a block and subsequently lyophilized. By splitting of the blocks after water removal to the desired thickness, water soluble fleeces were obtained, which by moistening behaved like sponges.

Patent claims

1. Collagen free cosmetic preparations, which can be obtained by cross-linking of swollen aqueous suspensions of chitosans and ß-(1,3) glucans with diisocyanates and/or dialdehydes.

2. Method for preparation of collagen free cosmetic preparations, by which swollen aqueous suspensions of chitosans and ß-(1,3) glucans are cross-linked with diisocyanates and/or dialdehydes, whereafter the water is removed.

3. Method according to claim 2, **characterised by** that chitosans with molecular weights in the area from 10 000 to 1 200 000 Daltons are used.

4. Method according to claim 2 and/or 3, **characterised by** that use is made of water soluble ß-(1,3) glucans, which are substantially free from ß-(1,6) linkages.

5. Method according to at least one of the claims 2 to 4, **characterised by** that use is made of diisocyanates with the formula (I)

O=CN-[X]-NC=O (I)

wherein X represents a linear or branced naphthenic or aromatic hydrocarbon residue with 1 to 12 carbon atoms.

6. Method according to at least one of the claims 2 to 5, **characterised by** that use is made of dialdehydes with the formula (II)

OHC-[Y]-CHO (II)

wherein Y represents a linear or branced naphthenic or aromatic hydrocarbon residue with 1 to 12 carbon atoms.

7. Method according to at least one of the claims 2 to 6, **characterised by** that hexamethylene diisocyanate and/or glutaric dialdehyde are used as cross-linking agents.

8. Method according to at least one of the claims 2 to 7, **characterised by** that alo polyols are used which are chosen from the group formed by glycerol, alkylene glycols, oligoglycerol mixtures of technical quality, methylol compounds, low alkyl glucosides, sugar alcohols, sugars and amino sugars.

9. Method according to at least one of the claims 2 to 8, **characterised by** that natural and/or synthetic fibres also are used.

10. Method according to at least one of the claims 2 to 9, **characterised by** that water is removed from the preparation by freeze-drying.

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PATENT

COMBINED DECLARATION AND POWER OF ATTORNEY

(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL, DIVISIONAL, CONTINUATION OR CIP)

As a below named inventor, I hereby declare that:

TYPE OF DECLARATION

This declaration is of the following type: *(check one applicable item below)*

- ☐ original
- ☐ design
- ☐ supplemental

Note: If the Declaration is for an International Application being filed as a divisional, continuation or continuation-in-part application, do not check next item; check appropriate one of last three items.

- ☒ national stage of PCT

Note: If one of the following 3 items apply, then complete and also attach ADDED PAGES FOR DIVISIONAL, CONTINUATION OR CIP.

- ☐ divisional
- ☐ continuation
- ☐ continuation-in-part (CIP)

INVENTORSHIP IDENTIFICATION

WARNING: If the inventors are each not the inventors of all the claims, an explanation of the facts, including the ownership of all the claims at the time the last claimed invention was made, should be submitted.

My residence, post office address and citizenship are as stated below, next to my name. I believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter that is claimed, and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

COLLAGEN-FREE COSMETIC PREPARATIONS

SPECIFICATION IDENTIFICATION

the specification of which: *(complete (a), (b) or (c))*

☐ (a) is attached hereto.

☐ (b) was filed on _____ as ☐ Serial No. _____ or
☐ Express Mail No. *(as Serial No. not yet known)* _____
and was amended on _____ *(if applicable).*

Note: Amendments filed after the original papers are deposited with the PTO that contain new matter are not accorded a filing date by being referred to in the Declaration. Accordingly, the amendments involved are those filed with the application papers or, in the case of a supplemental Declaration, are those amendments claiming matter not encompassed in the original statement of invention or claims. See 37 CFR 1.67.

☒ (c) was described and claimed in PCT International Application No. PCT/EP00/03762 filed on 26 April 2000.

ACKNOWLEDGEMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information, which is material to patentability as defined in 37, Code of Federal Regulations, § 1.56,

(also check the following items, if desired)

☐ and which is material to the examination of this application, namely, information where there is a substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent, and

☐ in compliance with this duty, there is attached an information disclosure statement, in accordance with 37 CFR 1.98.

PRIORITY CLAIM (35 U.S.C. § 119(a)-(d))

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

(complete (d) or (e))

☐ (d) no such applications have been filed.

☒ (e) such applications have been filed as follows.

Note: Where item (c) is entered above and the international application which designated the U.S. itself claimed priority check item (e), enter the details below and make the priority claim.

PRIOR FOREIGN/PCT APPLICATION(S) FILED WITHIN 12 MONTHS (6 MONTHS FOR DESIGN) PRIOR TO THIS APPLICATION AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. § 119(a)-(d)

COUNTRY (OR INDICATE IF PCT)	APPLICATION NUMBER	DATE OF FILING (day/month/year)	PRIORITY CLAIMED UNDER 35 USC 119
Germany	199 20 557.4	05 May 1999	☒ YES NO ☐
			☐ YES NO ☐

CLAIM FOR BENEFIT OF PRIOR U.S. PROVISIONAL APPLICATION(S) (35 U.S.C. § 119(e))

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:

PROVISIONAL APPLICATION NUMBER	FILING DATE

ALL FOREIGN APPLICATION(S), IF ANY, FILED MORE THAN 12 MONTHS (6 MONTHS FOR DESIGN) PRIOR TO THIS U.S. APPLICATION

_____

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Note: If the application filed more than 12 months from the filing date of this application is a PCT filing forming the basis for this application entering the United States as (1) the national stage or (2) a continuation, divisional, or continuation-in-part, then also complete ADDED PAGES TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR DIVISIONAL, CONTINUATION OR CIP APPLICATION for benefit of the prior U.S. or PCT application(s) under 35 U.S.C. § 120.

POWER OF ATTORNEY

I hereby appoint the following practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith *(list name and registration number)*.

Thomas F. Peterson, 24790; Richard J. Streit, 25765; Donald P. Reynolds, 26220; W. Dennis Drehkoff, 27193; Vangelis Economou, 32341; Brian W. Hameder, 45613; Valerie Neymeyer-Tynkov, 46956; Paul B. West, 18947; Joseph H. Handelman, 26179; Peter D. Galloway 27885; John Richards, 31503; Iain C. Baillie, 24090; Richard P. Berg, 28145

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☐ Attached, as part of this declaration and power of attorney, is the authorization of the above-named practitioner(s) to accept and follow instructions from my representative(s).

SEND CORRESPONDENCE TO:

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DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE(S)

Note: Carefully indicate the family (or last) name, as it should appear on the filing receipt and all other documents.

Full name of first joint inventor

Rolf (Given Name) ~~(Middle Initial or Name)~~ WACHTER (Family (or Last) Name)

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Inventor's signature X Rolf Wachter

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